



Space Flight Experiments to Measure Polymer Erosion and Contamination on Spacecraft

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ABSTRACT

Atomic oxygen erosion and silicone contamination are serious issues that could damage or destroy spacecraft components after orbiting for an extended period of time, such as on a space station or satellite. An experiment, the Polymer Erosion And Contamination Experiment (PEACE) will be conducted to study the effects of atomic oxygen (AO) erosion and silicone contamination, and it will provide information and contribute to a solution for these problems. PEACE will fly 43 different polymer materials that will be analyzed for AO erosion effects through two techniques: mass loss measurement and recession depth measurement. Pinhole cameras will provide information about the arrival direction of AO, and silicone contamination pinhole cameras will identify the source of silicone contamination on a spacecraft. All experimental hardware will be passively exposed to AO for up to two weeks in the actual space environment when it flies in the bay of a space shuttle. A second set of the PEACE Polymers is being exposed to the space environment for erosion yield determination as part of a second experiment, Materials International Space Station Experiment (MISSE). MISSE is a collaboration between several federal agencies and aerospace companies. During a space walk on August 16, 2001, MISSE was attached to the outside of the International Space Station (ISS) during an extravehicular activity (EVA), where it began its exposure to AO for approximately 1 1/2 years. The PEACE polymers, therefore, will be analyzed after both short-term and long-term AO exposures for a more complete study of AO effects.

I. INTRODUCTION

Atomic oxygen erosion in the low Earth orbit (LEO) environment is a serious problem the space program faces today. Atomic oxygen is produced in the upper atmosphere when the sun's short wavelength ultraviolet (UV) rays photo-dissociate diatomic oxygen. Although atomic oxygen is the predominant species in LEO (below $\approx 1,000$ km), these neutral oxygen atoms have mean free paths on the order of 10^4 m at 400 km, resulting in extremely low probabilities of re-association. When a spacecraft is orbiting in LEO at 7.7 km/sec, the AO atoms ram into the

spacecraft with enough energy (average of ≈ 4.5 eV) to break chemical bonds (Ref. 1). The functional polymers on the craft's surface oxidize and, because the oxidation product is most often a volatile species, the polymers literally erode away (Ref. 1). In addition, the volatile products can redeposit and contaminate other spacecraft surfaces. See Figure 1 for a depiction of the effects of AO erosion.

Polymers are regularly utilized on spacecraft surfaces, for example the Kapton solar array blankets on the ISS and the Teflon FEP (fluorinated ethylene propylene) insulation covering the Hubble Space Telescope, because of their low density, flexibility, and optical or electrical properties. When AO erodes these polymers, it increases the danger of structural failure. Atomic oxygen erosion can also reduce the emittance of thermal control materials, which can cause a damaging increase in temperature in the spacecraft.

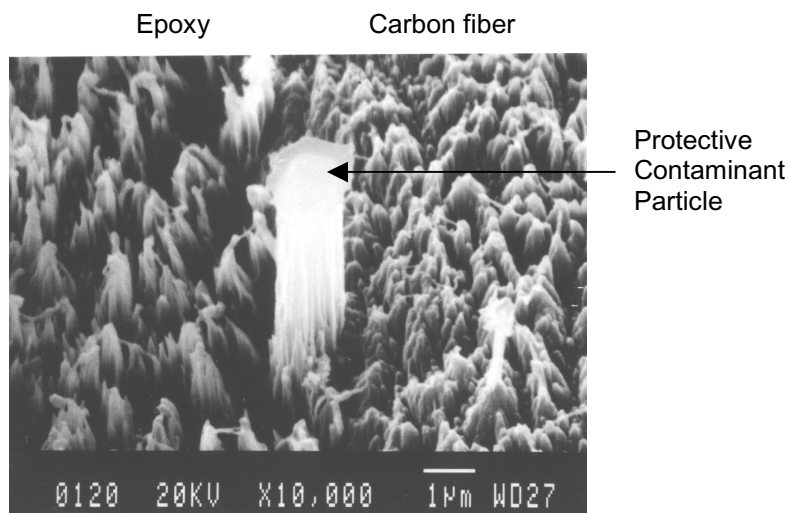


Figure 1. Image of AO erosion of a graphite epoxy composite sample exposed to an AO fluence of 2.3×10^{20} atoms/cm². This sample was flown as part of the Environmental Oxygen Interaction Materials (EOIM-III) experiment aboard STS-46 (Ref. 2).

Silicone contamination is considered one of the most significant problems for a spacecraft like the ISS. Products that outgas from silicone components, or are produced through UV interaction, can collect on other surfaces of the spacecraft and eventually form a thick layer of contaminant, which is then fixed and darkened by further UV exposure. When silicone is exposed to AO, both the original silicone and the silicone contaminant layer can oxidize and form silica, with a glassy surface (Ref. 3). In addition, AO likely causes additional outgas products to occur, resulting in greater amounts of contamination. On the Russian space station MIR, contamination was up to 1 micron thick and affected everything from its solar panels to handrails used by astronauts and cosmonauts during space walks (Ref. 4). There is currently no technique to identify the source of silicone contamination on a spacecraft.

The Polymer Erosion And Contamination Experiment Project will study the effects of AO through spaceflight experiments and involves collaboration between professionals at NASA Glenn Research Center and students from Hathaway Brown High School. PEACE consists of the development of two novel space flight experiments to address the problems of AO erosion and silicone contamination: PEACE and MISSE PEACE Polymers (also called PEACE on MISSE). PEACE will be exposed to the LEO environment for a short-term exposure of about ten days, while the MISSE PEACE Polymers are currently being exposed to LEO for an ultimate period of twelve to eighteen months. Both experiments will provide a better understanding of

the effects of AO erosion on different polymers. The resulting data will be shared with the space community and will contribute to the development of materials durable to AO erosion.

II. PEACE (Polymer Erosion And Contamination Experiment)

PEACE will fly in a Get Away Special (GAS) Canister that will be mounted in the payload bay of a space shuttle orbiter flown in LEO. It is a short-duration passive experiment and will be exposed to the LEO environment for approximately ten days. PEACE has two main objectives: to measure the AO erosion yields of 42 different, carefully characterized polymer materials, and to validate a technique for the identification of sources of silicone contamination through the use of contamination pinhole cameras. An engineering drawing of the PEACE experiment is shown in Figure 2.

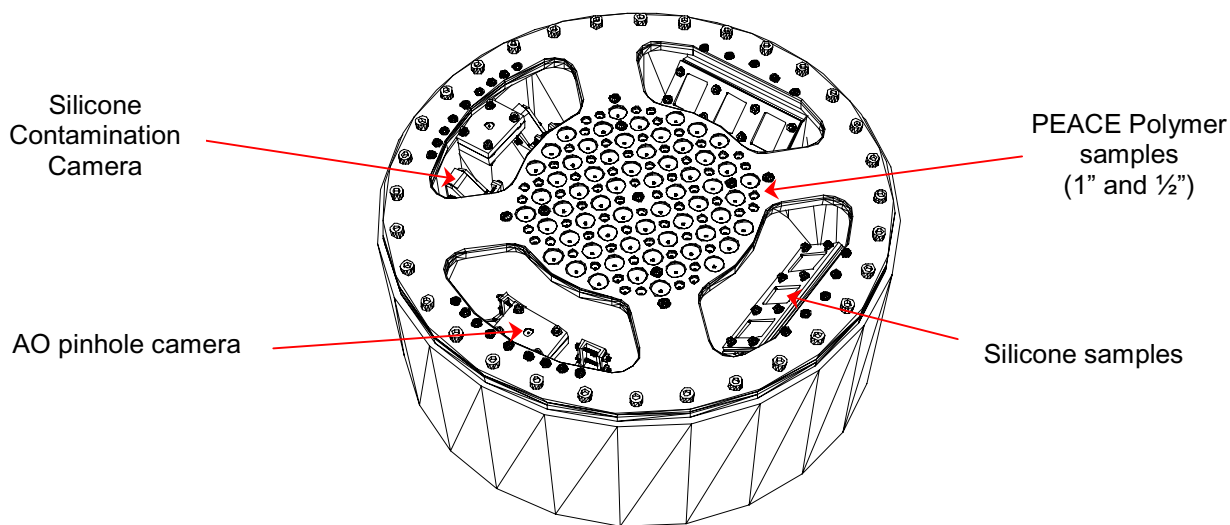


Figure 2. Three-dimensional engineering drawing of PEACE.

A. Polymers for Erosion Yield Determination

The AO erosion yields of the polymers will be determined using two different techniques: mass loss measurements, which is the traditional method, and recession depth measurements. Hygroscopic materials can have large weight variations because they absorb moisture, so each of the polymer's dehydrated masses will be obtained pre- and post-flight. However, the mass loss of polymers exposed for a short time can be within the margin of error, so a new technique based on recession depth will also be utilized. This method requires each of the polymers to be sprayed with either a salt solution or mica dusted, selectively protecting the polymers from erosion. Post-flight, the recession depth measurements will be taken using an atomic force microscope (AFM) after removal of the protective particles (Ref. 2).

The experiment consists of 43 different polymer materials: 42 different polymer materials for characterization, and Kapton HN fluence witness samples. Each of the 42 polymers flown will have one 1 in. (2.54 cm) sample and one 1/2 in. (1.27 cm) sample on the experiment. Together with 44 Kapton HN witness samples, a total of 124 polymer erosion samples will be flown: 60 large (1in. diameter) samples for mass loss determination, and 64 small (1/2 in. diameter) samples for recession depth measurement. Refer to Figure 2 for the sample layout for the 128 polymer samples. Kapton HN has a well-characterized erosion yield in LEO, so its

erosion will be used to determine the final AO fluence for the experiment. The 42 polymer materials were chosen because they have a variety of bonding types and AO erosion yields. Even though it is undesirable to use some of these polymers for spacecraft applications, their chemistry can be used to determine the AO durability of new materials, without need for expensive space exposure testing. The specific polymers to be flown in the PEACE shuttle flight experiment are listed in Table 1.

Table 1. PEACE Polymers List

PEACE #	Material	Abbrev.	Trade Names
1	Acrylonitrile butadiene styrene	ABS	Cycolac
2 s	Cellulose acetate	CA	Clarifoil; Tenite Acetate; Dixel
2m	Polyethylene oxide	PEO	Alkox E-30
3	Polybutylene terephthalate	PBT	GE Valox 357
4	Chlorotrifluoroethylene	CTFE	Neoflon CTFE M-300; Kel-F
5	Crystalline polyvinylfluoride w/white pigment	PVF	White Tedlar TW10B53
6	Diallyl diglycol and triallyl cyanurate	ADC	CR-39, Homalite H-911
7	Epoxide or epoxy	EP	Hysol EA 956
8	Perfluoroalkoxy copolymer resin	PFA	Teflon PFA CLP (200 CLP)
9	Tetrafluorethylene-ethylene copolymer	ETFE	Tefzel ZM
10*	Cellulose Nitrate	CN	Celluloid
11	High temperature polyimide resin	PI	PMR-15
12	Fluorinated ethylene propylene	FEP	Teflon FEP (round robin)
13	Graphite	PG	Pyrolytic Graphite
14	Halar ethylene-chlorotrifluoroethylene	ECTFE	Halar
15	Polyimide (BPDA)	PI	Upilex-S
16*	Polyimide	PI	Kapton E
17	Polyimide (PMDA)	PI	Kapton H
18 s	Ultra High Molecular Weight Polyethylene	UHMWPE	
18 m	Poly-(p-phenylene terephthalamide)	PPD-T	Kevlar 29 fabric
19	Polyamide 6 or nylon 6	PA 6	Akulon K; Ultramid B
20	Polyamide 66 or nylon 66	PA 66	Maranyl A; Zytel
21	Polyacrylonitrile	PAN	Barex 210
22	Polybenzimidazole	PBI	Celazole PBI
23	Polycarbonate	PC	PEEREX 61 (P61)
24	Poly(p-phenylene-2,6-benzobisoxazole)	PBO	Balanced Biaxially Film
25	Polyethylene	PE	
26	Polyetheretherketone	PEEK	Victrex PEEK 450
27	Polyethylene terephthalate	PET	Mylar A/200
28	Polyimide	PI	LaRC CP1 (CP1-300)
29	Polymethyl methacrylate	PMMA	Plexiglas; Lucite; Acrylite (Impact Mod.)
30	Polypropylene	PP	Type C28
31	Polyoxymethylene; acetal; polyformaldehyde	POM	Delrin (Natural)
32	Polystyrene	PS	Trycite 1000/Trycite Dew
33	Polysulphone	PSU	Thermolux P1700-NT11; Udel P-1700
34	Polytetrafluoroethylene	PTFE	Chemfilm DF 100
35	Polyurethane	PU	Dureflex PS 8010
36	Polyvinylidene fluoride	PVDF	Kynar 740
37	Polyvinyl fluoride	PVF	Tedlar TTR10SG3
38	Polyetherimide	PEI	Utem 1000
39	Amorphous Fluoropolymer	AF	Teflon AF 1601
40	Polyphenylene isophthalate	PPPA	Nomex Aramid Paper Type 410
W	Polyimide (PMDA)	PI	Kapton HN

* Different than MISSE numbers

Two AO pinhole cameras will determine the direction from which the AO came. Within the cameras silver-coated glass hemispheres will oxidize when impacted by AO, providing a “map” of the AO arrival direction. The pinhole cameras will also provide information about AO shadowing effects on the samples. The AO pinhole cameras are shown in Figure 2.

B. Silicone Contamination Camera and Samples

The silicone contamination cameras are the second part of the PEACE experiment. Four pinhole cameras, one of which can be seen in Figure 3, will be flown to validate a method for identifying the source of silicone contamination. The silicone contamination camera is a newly developed concept that has not been tested in the actual space environment. It will provide an image of the source of silicone contamination on a glass plate behind a pinhole window. The camera is designed to allow the conversion of silicone contamination to silica on the glass plate through AO oxidation, thus fixing and enhancing the contamination build-up and simulating what occurs on spacecraft. This method is expected to verify a technique that can be used in future missions to provide an image of the contaminant source on spacecraft in LEO.

Four silicone materials will be flown on three sample slides opposite two pinhole cameras. Three slides will be open to AO through an opening in the top plate of PEACE (see Figure 2), and an identical three will be shadowed and protected from AO erosion. The objective of having both AO exposed and AO shaded silicone samples is to test if AO will enhance the amount of contaminants departing the silicone samples. Contaminants (volatile products of the silicones) will enter through the orifice plate of the camera and land on a glass contamination deposition plate within the camera, forming a reverse image of the contaminant source. Because the glass plate will also be exposed to AO, the silicone contamination will oxidize and fix the contamination image into glassy silica. The silicone samples that are not exposed to AO are expected to produce a thinner contaminant deposit on the deposition plate than those that are exposed to AO. Contaminant thickness will be analyzed post-flight using scanning variable angle spectroscopic ellipsometry (VASE).

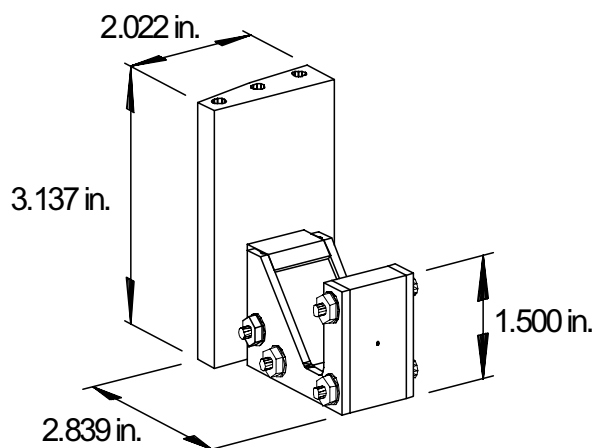


Figure 3. Assembled view of the silicone contamination pinhole camera.

The four silicone samples being tested are DC 93-500, Kapton tape adhesive (Polymethylphenylsiloxane (PMPS) or Polydimethylsiloxane (PDMS)), RTV-560, and Dimethylethoxysilane (DMES). These four silicones have practical applications on most

spacecraft: DC 93-500 is a common solar cell adhesive, Kapton tape is an adhesive used throughout spacecraft, RTV-560 is a shuttle tile adhesive, and DMES is a water-proofing silicone for shuttle tiles. Because all of these silicones are used concurrently on spacecraft, they are all plausible but uncharacterized sources of contamination.

III. PRE-FLIGHT EXPERIMENTATION FOR PEACE

A. Pre-flight Sample Fabrication

A.1. Polymer Samples. Polymer samples in the form of thin sheets were obtained directly from manufacturers or suppliers. Samples were fabricated by forcing a circular Osborne double-bow Punch, either 1 in. (2.54 cm) or ½ in. (1.27 cm) in diameter, through a thin sheet of the polymer using an Arbor press. The subsequent polymer samples were stored carefully until needed. It was imperative that the polymers be kept clean, particularly from fingerprints. Salt from skin could have accumulated on the polymer samples and influenced the flight data.

A.2. Silicone Samples. Practice silicone samples were made in the same manner flight samples will be made. The DC 93-500 and RTV-560 silicone flight samples will be applied to stainless steel substrates measuring 2 in. × 1 3/8 in. (5.08 cm x 3.49 cm). To prepare the substrate for DC 93-500, a layer of 2-mil (0.005 cm) thick tape was applied to its edges. The base and curing agent of the silicone were mixed together in a ten to one ratio and poured onto the substrate. A microscope slide was used to scrape off the excess silicone, and the sample was cured at 65 °C for 4 hours. After it was cured, the tape was removed to leave a 2-mil (0.005 cm) thick silicone sample. A similar technique was used for making practice samples of RTV-560, while following its suggested procedure. However, for the RTV-560 samples a primer needed to be applied first, and both the primer and silicone were added to one half of the substrate, leaving the other half for the Kapton tape samples.

The desired thickness for the RTV-560 and DC 93-500 silicones is 2-mil (0.005 cm). To achieve this thickness, as mentioned, a 2-mil (0.005 cm) thick tape was applied around the edges, so that when the silicone was applied to the slide and spread flat with a microscope slide edge, the thickness of the remaining silicone was the same as the tape depth. This method provided results ranging from 1.4375 mil (0.0036 cm) to 3.138 mil (0.0078 cm) for the RTV-560 (on different slides). While these results need to be improved upon, an even thickness of silicone on each individual substrate was achieved. The practice samples for the DC 93-500 varied greatly in thickness, ranging from 2.83 to 4.17 mils (0.0070 cm to 0.0104 cm). The variation could be ascribed to the fact that these were the first samples to be made and indicates that better lab techniques must be used to apply this silicone to provide thinner and more uniform silicone samples.

B. Mass Loss Erosion Measurement Technique

B.1. Mass Loss Erosion Yield Technique. The most common technique for determining the erosion yield of flight samples is through mass loss measurements, obtained by taking mass measurements of the flight sample pre- and post-flight. The erosion yield of the sample (E_s) in cm³ of polymer eroded per impacting atom is calculated through the following equation (Ref. 6):

$$E_s = \frac{\Delta M_s}{(A_s \rho_s F)} \quad (1)$$

where

ΔM_s = mass loss of the flight sample (g)

A_s = surface area of the flight sample exposed to atomic oxygen attack (cm^2)

ρ_s = density of sample (g/cm^3)

F = fluence of atomic oxygen (atoms/cm^2)

Because Kapton HN has a well-characterized erosion yield in LEO ($3.0 \times 10^{-24} \text{ cm}^3/\text{atom}$), the AO fluence for the mission will be determined from Kapton HN's mass loss (using the same equation for fluence above). The mass loss measurement technique will be used to determine the AO erosions of polymers on both the PEACE and MISSE PEACE Polymers experiments.

B.2. Rehydration Curves. The amount of moisture in the atmosphere affects the mass of certain polymers. Many polymers are hygroscopic and absorb water from the air, so that if they are measured on two different days, the measurements could vary because of fluctuating humidity. Rehydration curves, which show an increase of a dehydrated sample's mass over time, are being conducted for all 43 polymer samples to ascertain the degree to which the polymers are affected by moisture in the air.

Rehydration curves are essential because they provide information on how long a sample must be under vacuum before it becomes completely dehydrated, and how quickly samples absorb moisture. This information will be used to determine how long each sample must be dehydrated before pre-flight mass measurements can be taken. Each polymer acquires moisture at a different rate; for example, Kapton takes on moisture very quickly, whereas graphite is thick and absorbs moisture slowly. In an ideal curve, a sample's mass rises at an even interval until its mass levels off, meaning it has taken on its maximum amount of moisture.

To determine a rehydration curve, a sample is put in a vacuum desiccator for at least four days in an attempt to completely dehydrate it. The sample is then taken out, weighed immediately to provide a near-completely dehydrated mass measurement, and weighed periodically at 1, 2, 3, 8, 18, 38, 68, and 128 minutes past the time of initial exposure. The data is graphed and shows how the sample gains moisture. An example is shown in Figure 4.

C. Recession Depth Measurement Technique

C.1. Technique Development. A new technique based on recession depth measurements has been developed and will provide more accurate erosion yield data for low fluence exposures or very low erosion yield samples (Ref. 2). Dry salt crystals have been found to protect the underlying areas in direct contact with a polymer against AO erosion, so the PEACE ½ in. (1.27 cm) flight samples will be sprayed with a saltwater solution. They will be sprayed using a method currently being developed to selectively protect each polymer from AO attack in space. After flight, the salt particles will be removed and the erosion depth will be determined using AFM (see Figure 5). Some of the selected polymers react negatively because of exposure to water. If condensation buildup is a problem (see section C.2), these samples will be coated with mica dust instead, which has the same protection against AO but does not require water contact for application or removal.

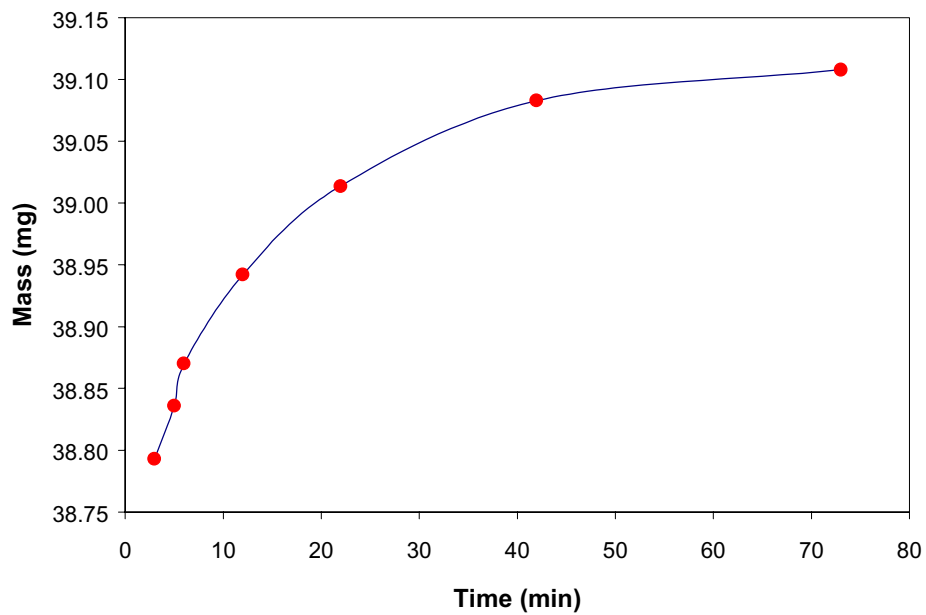


Figure 4. Rehydration curve for 2-mil thick Kapton HN dehydrated for 5 days.

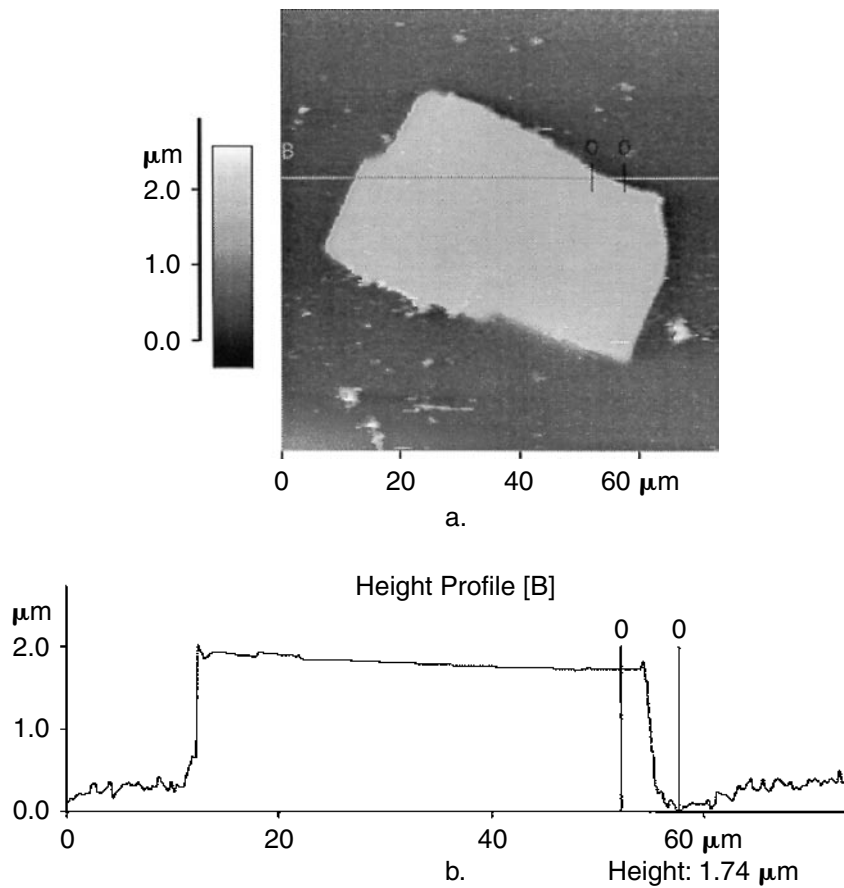


Figure 5. Example of recession depth analysis using AFM: a.) topographical image of a salt-particle protected island after AO erosion, and b.) profile of line trace indicated in 5a of protected island after post-flight salt removal.

As part of the development of this selective protection recession technique, data from a salt-sprayed sample that was flown on an earlier shuttle experiment is being used to further develop and improve the technique. Many experiments have been conducted so far to characterize the salt morphology and develop a technique for repeatable application of small, square salt crystals in good distribution. An acceptable method is currently being improved to achieve a repeatable method of application. Several variations of application have been tested, including different atomizers, distance of initial spray and sample, type of salt (iodized vs. non-iodized), type of water (distilled vs. tap), temperature of solution, and movement or immobilization of sample through the salt-solution cloud. See Figure 6 for variations that can occur in salt crystals. Using the conclusions of these experiments, the latest method is spraying a salt spray cloud through a hole from one end of a box approximately one meter long, and swooping the samples through the cloud, inside the box. This creates a controlled environment, which allows relatively similar results to be achieved repeatedly. The best solution is saturated with non-iodized salt in distilled, room temperature water.

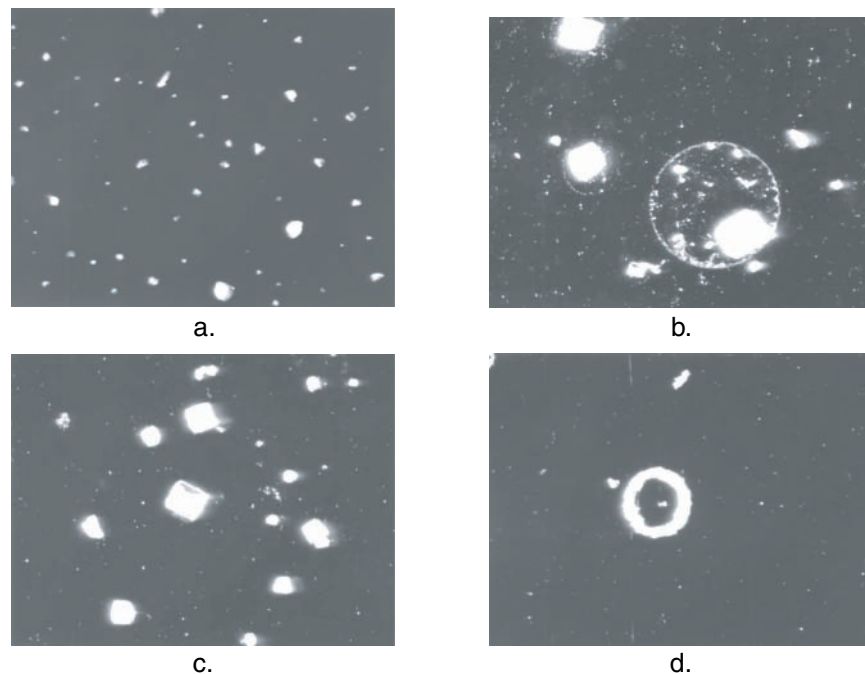


Figure 6. Optical micrographs of different types of salt particles formed on a Kapton HN substrate during salt-spraying: a. small irregular shaped particles, b. salt rings around large (roughly cubic) crystals, c. large cubic and irregularly shaped crystals and d. salt residue in doughnut shapes (Ref. 2)

C.2. Condensation Tests. After PEACE has been loaded on the shuttle prior to flight, a potential problem that may occur is condensation from the air accumulating on the polymers, which may have a negative effect on the salt crystals. Testing has shown that salt crystals act as nucleation sites for condensation and that they will dissolve as water builds up. The resulting saltwater may spread (or "wet down") on the sample depending on the interfacial surface energy, or surface tension, between the three interfaces: the polymer surface and the air, the saltwater and the air, and the saltwater and the polymer. Therefore, the interfacial surface energy will affect the shape of the saltwater bead. If the saltwater spreads out as a thin layer on the polymer, many smaller salt crystals that are potentially touching may form as the saltwater dries. This is not desirable because individual isolated salt particles are needed for the AFM-recession depth.

In anticipation of this problem, freezer-condensation tests were conducted for each of the polymers, in which condensation was purposely allowed to accumulate on salt-sprayed samples, and the effects of the condensation on the salt particles were observed. These tests indicate which samples would have negative effects because of condensation and therefore should be protected with another form of protective particles such as mica dust.

The samples were sprayed with a salt solution, and specific salt crystals were photographed using an Olympus SZH stereo microscope. The samples were then placed in a freezer for a minimum of 3-4 hours to allow the samples to get cold. After being taken out, they were briefly exposed to room temperature air, which caused condensation to nucleate at the salt particles. Photographs of the same crystals were taken of the post-freezer samples after the salt solution had dried. The pre- and post-freezer pictures were compared and analyzed to determine how condensation affected the salt. Figure 7 is an example of a typical freezer test sequence.

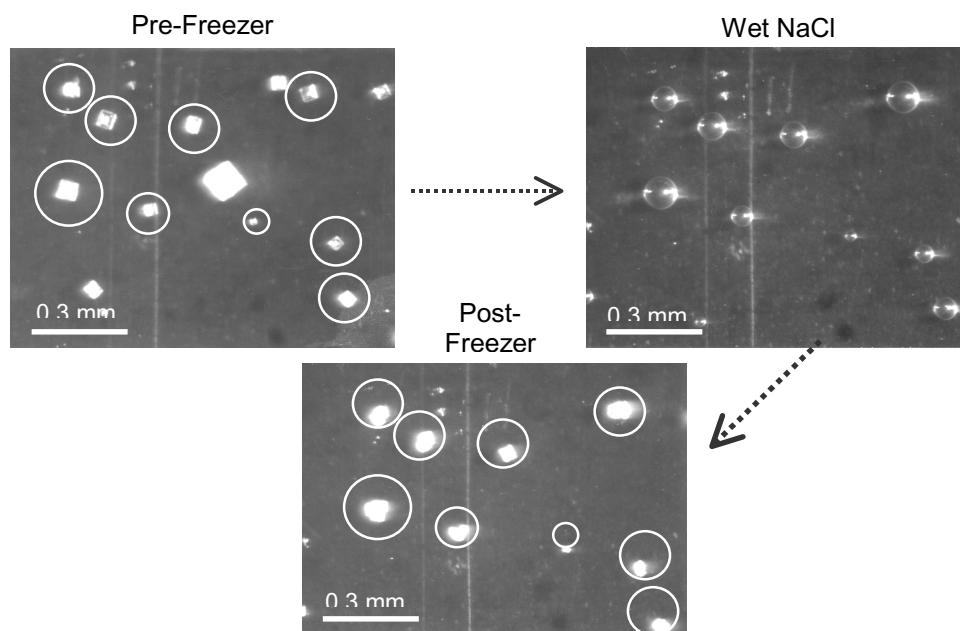


Figure 7. A chronological series of optical micrographs taken of the polymer ethylene-chlorotrifluoroethylene (ETCFE), also known as Halar, typical of a freezer-condensation test.

It was concluded that condensation of salt crystals is not a problem for most polymers. On some polymers, the condensation changed the shape of the salt crystals, making them less cubic (such as seen in Figure 7). Although a cube is the optimal crystal shape, in most cases the crystals were not altered enough that they would be insufficient at protecting the polymer. The salt crystals on the polymer Barex were broken into groups of smaller crystals by the condensation. These small groups of crystals may not be adequate for use of the AFM technique, so this polymer will have to be mica dusted. In some cases, the condensation caused faint rings to appear around the crystals. Further experimentation determined these rings are undesirable, but crystals with salt rings can still be used for erosion determination, as discussed in section C.3. The only other polymers which may need to be mica dusted rather than salt sprayed are those which did not react well to the salt spray and immediately wetted out, and those that dissolve in water, such as polyethylene oxide.

C.3. Salt Rings. During salt-solution application to the samples, rings of salt residue sometimes form around salt crystals. A particular concern was whether the area within the rings was protected or partially protected from AO. If the area inside the salt ring was partially protected, then those crystals could not be used for recession depth analyses. An experiment was conducted in which a salt-sprayed sample was exposed to AO in a LEO-simulating plasma asher. The salt was then removed and the sample was observed using a scanning electron microscope (SEM). See Figure 8 for images of salt rings before and after AO exposure.

It was determined that the salt ring itself protected the polymer from erosion, but the area within the ring was unaffected by it. Therefore, crystals with salt rings, though undesirable, are tolerable for obtaining flight data.

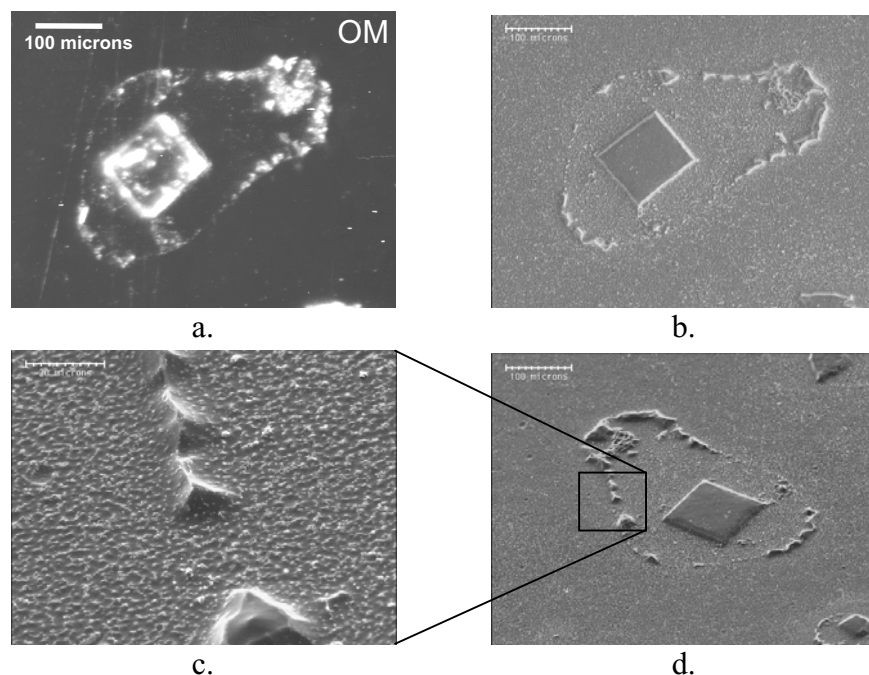


Figure 8. Set of images of a cubic salt crystal surrounded by a salt ring: a. optical micrograph taken prior to AO exposure (salt crystal present), b. SEM image taken after AO exposure (salt crystal removed), c. close up of the salt ring section indicated in d, and d. SEM image taken after AO exposure at $\approx 30^\circ$ tilt angle.

C.4. AFM Technique Development. Post-flight, the salt-sprayed samples will be analyzed using an AFM. The AFM will be used to measure the step-height of the region protected by a salt crystal from the eroded surface of the polymer to the eroded valley. An AFM can be operated in two common topographical data acquisition modes: contact and non-contact. In contact mode, a cantilevered tip directly contacts the surface of the sample, while in non-contact mode, the tip hovers approximately 50 Å above the surface. Contact mode imaging, which is much easier to operate than non-contact mode imaging, could damage the delicate erosion texture thus affecting the erosion analysis. An experiment was conducted using a Park Scientific AutoProbe LS AFM imaging an LEO exposed salt-sprayed Kapton sample using both contact and non-contact modes, and the results were compared. It was concluded that contact imaging does not affect the eroded surface and can be used post-flight to analyze the PEACE polymers. Details of the AFM data acquisition comparison test and flight sample are reported by de Groh et al in Reference 2.

C.5. Uncertainty Analyses. Small cubic salt crystals are desirable because the error for measurement increases with the crystal height, and square crystals provide ideal protection from AO because the flat bottom surface is in intimate contact with the polymer. For low fluence missions, the AFM method is the most accurate way to measure the AO erosion of a sample when it is protected by very small crystals (Ref. 2). But mass loss measurements become a more accurate method to determine erosion yield once the crystals for recession depth reach a height of 17 microns (Ref. 2). Figure 9 shows the different probable errors as a function of AO fluence for the mass loss and AFM recession techniques using a small (10 μm thick) crystal.

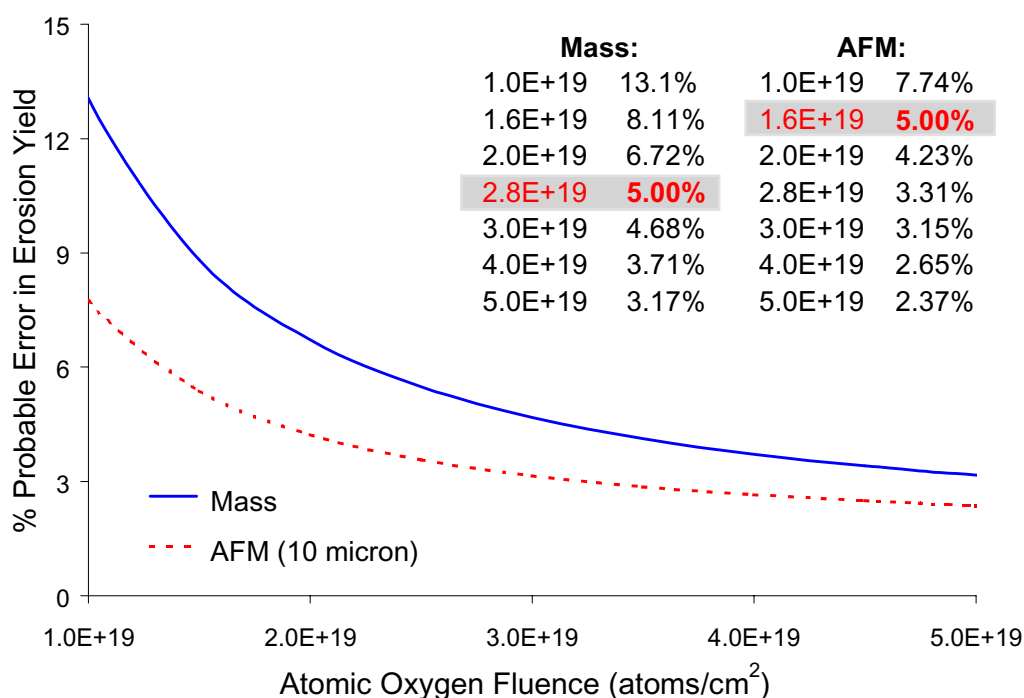


Figure 9. Comparison of the percent probable error in the atomic oxygen erosion yield as a function of AO fluence for the mass loss technique and the AFM recession technique (for 10 μm thick protective particles) (Ref. 2).

For example, at a low AO fluence such as 2×10^{19} atoms/cm², there is a 4.23% probable error in erosion yield when the sample is protected by a 10 μm thick salt crystal and analyzed by the AFM method, a 6.72% error when analyzed with mass loss measurements, and a 9.54% error when it is protected by a 25 μm thick crystal and analyzed with the AFM method (Ref. 2). For protective particles less than 50 μm thick, as the AO fluence increases, the percent error for both methods becomes much lower and closer together until the distinction in accuracy between them is insignificant for higher fluences. For example, at an AO fluence of 5×10^{20} atoms/cm², there is a 1.92% probable error when the sample is protected by a 50 μm thick salt crystal and analyzed by the AFM method, and a 1.85% error when analyzed with mass loss measurements.

D. Gold Standards

Over time, calibrations of the balances used to weigh the mass loss samples pre-flight may change. Even if the margin of error was small since the mass losses are also small, it could result in overall misinterpretation of the mass loss of samples due to AO erosion, particularly during a short-exposure experiment with minimal erosion. To solve this problem, gold pellets

were weighed on each of the balances used for pre-flight analysis because gold's mass will not fluctuate over time. The pellets were selected based on mass: there are ten weight standards, and each represents an interval of mass from the lightest to the heaviest polymers. They were all weighed on three different days, and the average of these weights was obtained. Post-flight, the gold pellets will be weighed on the balances and the same measurements will be taken. The average of the post-flight measurements will be compared to the pre-flight measurements; any fluctuation of mass measurement of samples will be corrected for in the analysis of post-flight samples.

E. PEACE Database

A PEACE database has been created to record all of the pre-flight and post-flight data and results. The database was designed using Microsoft Access software. Each of the 42 samples has two sheets, one for the sample that will be flown in space and a second one for a backup sample. The first part of the database contains all of the information by which the sample can be identified, including: identification number, sample size, sample description, material abbreviation, trade name, supplier, polymer formula, and the structure of the polymer. The next part of the database was organized to catalog the results from measurements and dehydration. This section includes: pre-flight thickness, density, sample orientation and sample scribe identification, protection method, dehydration mass before and after, dehydration information, erosion depth, post-flight erosion yield, and finally a section for special concerns about that specific sample.

IV. MISSE PEACE POLYMERS

The MISSE is a materials environmental exposure experiment that is managed by NASA Langley Research Center and contains samples from various NASA centers, the U.S. Air Force and private industry. It will test the durability of hundreds of materials in the space environment. MISSE consists of Passive Experiment Containers (PECs) to be exposed to AO and solar radiation or solar radiation with no AO exposure for approximately 1-year (PEC 1 and 2) and 3-year exposures (PEC 3 and 4) (Ref. 8).

A second set of the same polymers to be flown in the shuttle experiment PEACE are being flown as part of MISSE and are collectively called the MISSE PEACE Polymers (Ref. 6). They are being exposed to AO and solar radiation over a period of approximately 1 1/2 years, which began when MISSE was mounted outside of the ISS on August 16, 2001 during an extra-vehicular activity (EVA). There are a total of 41 MISSE PEACE Polymers, including 2 Kapton H witness samples. Three polymers to be flown on PEACE are not part of the MISSE PEACE Polymers set (cellulose nitrate, Kapton E, Ultra high molecular weight polyethylene). The MISSE PEACE Polymers experiment will only study the erosion yields of the polymers by mass loss evaluation, because the AO fluence should be high enough that mass measurements should provide low uncertainties in the erosion yield data. Through the results from both PEACE and MISSE PEACE Polymers, information on the polymers will be available for both short- and long-term exposure.

A. MISSE PEACE Polymers Sample Layering

Because the polymers will be on the ISS for 1 1/2 years or more, one thin polymer sample may not withstand the large amount of AO erosion, depending on the film thickness and the erosion yield of the polymer. Therefore, instead of one sample, a stack of samples for each polymer was placed in the holder. To decide how many samples to put in a stack, the erosion yield of each polymer was determined using previous flight data or estimations based on the polymer chemistry. The AO fluence for 1.5 years was computed to be 4.55×10^{21} atoms/cm², so from the polymer's erosion yield and this AO fluence, the estimated thickness loss after 1.5 years was determined using the equation:

$$\text{Erosion yield (cm}^3/\text{atom)} \times \text{Fluence (atoms/cm}^2\text{)} = \text{Thickness loss (cm)} \quad (2)$$

The estimated thickness loss was divided by the thickness of a single polymer layer to determine the number of samples that should be weighed together pre-flight. Behind these weighed samples, additional un-weighed samples were stacked in case the experiment is not retrieved from the ISS after 1.5 years. This assures that there is enough of the polymer to withstand the space environment for up to three years. These stacked samples are left un-weighed because if the starting mass is larger, there will be a bigger difference in error of the percent mass loss of the sample. Only the remaining weighed samples will be re-weighed after flight.

For example, the estimated erosion yield for polyvinyl fluoride (PVF) is 3.40×10^{-24} cm³/atom. The predicted thickness lost after 1.5 years is 6.1 mils (0.0153 cm), and 12.2 mils (0.031 cm) for 3 years. The thickness of one flight sample of PVF is 1.0 mil (0.0025 cm), so 7 individual samples needed to be stacked together and weighed, and 6 samples stacked behind it, to ensure that there will still be polymer material left after 3 years in space.

B. MISSE Flight Sample Preparation

B.1. Vacuum Baking of Flight Sample. According to the American Society for Testing and Materials (ASTM) standards, samples flown in space must be evaluated to ensure that they do not outgas (release volatile products) excessively, because that leads to the possible contamination of other samples. Under ASTM E-595 (Ref. 7), a sample is placed in a vacuum at 125°C for 24 hours, and a cooler sample (25°C) is placed next to it, which is used to determine how much of the sample's lost mass will condense on other samples (Ref. 7). Historically, it is required that less than 1.00% Total Mass Loss (TML) and less than 0.10% of Collected Volatile Condensable Materials (CVCM) should be produced to be acceptable for spacecraft use. The MISSE materials needed to meet these criteria. While most of the samples had already been tested and met the standards, ten did not, so these ten samples were vacuum baked to eliminate volatile materials before flight (Ref. 6). Pre-flight, every sample was either approved by prior results or vacuum baked sufficiently to prevent outgassing problems.

B.2. Sample Weighing and Flight Hardware Loading. Pre-flight mass measurements were obtained for dehydrated sample stacks. All stacks were dehydrated in a vacuum desiccator for a recorded time that was at least 4 days. The stacks were then weighed on a Mettler Balance or Sartorius Balance. One stack was weighed at a time, and the other samples were immediately put back under vacuum after a stack was removed from the desiccator. Each stack was weighed three times, and the average of these three readings was determined.

Final flight preparations took place after sample weighing was completed. The MISSE PEACE Polymer samples were loaded into a sample tray in a clean room environment, as seen in Figure 10. Ultraviolet lights were used to identify dust on the samples, and the dust was removed with a slow nitrogen gas flow or tweezers. Figure 11 is a photograph of the sample tray ready for flight, with the polymers identified. After the PEACE Polymer tray was loaded, the tray was integrated into MISSE PEC 2, as shown in Figure 12.



Figure 10. PEACE Team with loaded MISSE PEACE Polymer samples in clean room.

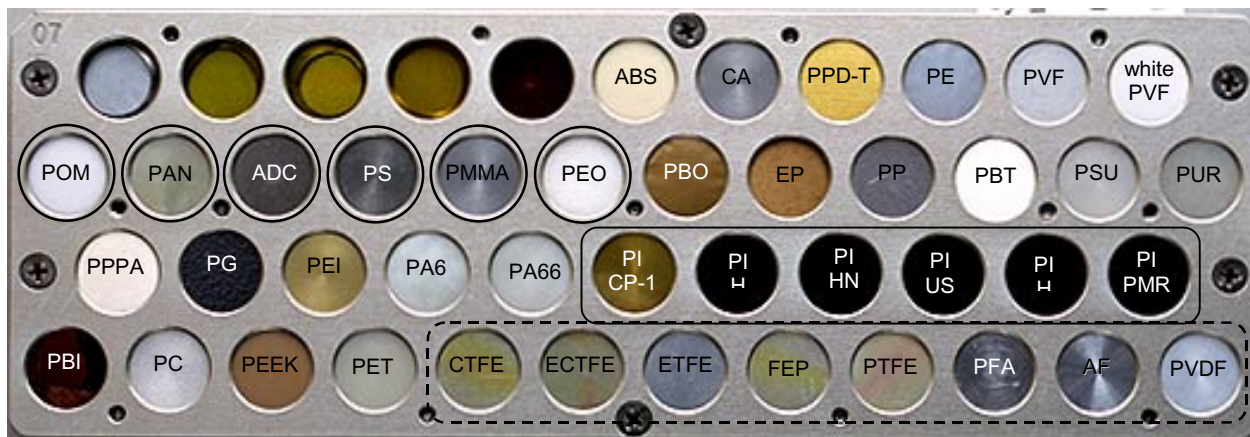


Figure 11. Forty-one MISSE PEACE Polymers loaded into MISSE sample tray E5. Samples with circles (POM through PEO) have expected high erosion yields, samples within the solid line block are from the polyimide family and samples within the dashed line block are from the fluoropolymer family.



Figure 12. MISSE PEC 2 with Tray 1 on the right (atomic oxygen and solar exposure tray) and Tray 2 on the left (solar exposure only tray) (Ref. 8).

C. Launch of MISSE on STS-105

During shuttle mission STS-105 to the ISS, the two MISSE PEC's were attached to the exterior of the ISS during an EVA on August 16, 2001. Figure 13 shows attachment of MISSE PEC 2 to the exterior of the Quest Airlock by astronaut Patrick Forrester. Currently, the MISSE polymers are being exposed to LEO conditions until their retrieval during another EVA scheduled for the fall of 2002.



Figure 13. During a spacewalk on August 16, 2001, astronaut Patrick Forrester installs MISSE PEC 2 on the ISS Quest Airlock, the first external experiment on the ISS hull (NASA photo STS105E5302). The MISSE PEACE Polymers are visible in this photograph.

V. CONCLUSIONS

Two space flight experiments are being jointly conducted by NASA Glenn Research Center and Hathaway Brown School to study the effects of AO erosion on spacecraft materials, a significant problem to the space program. PEACE consists of 43 polymer materials and pinhole cameras and will provide information on the erosion yields of polymers and on silicone contamination of spacecraft components. The erosion yields will be determined using two techniques based on either mass loss or recession depth, both of which have been reviewed in this paper. PEACE will be exposed to LEO conditions from the shuttle bay of an orbiter. A second set of the PEACE polymers is part of the experiment MISSE. The MISSE PEACE Polymer samples have been fabricated, characterized, and loaded into experimental hardware in a clean room. They are now being exposed to AO on the exterior of the ISS, and are scheduled to be retrieved for analysis in the fall of 2002.

Various pre-flight laboratory tests have been necessary for characterization of the polymers and experiment development for both flight experiments. Tests included studying salt particle application techniques, the effects of condensation on salt-sprayed samples and issues related to salt rings. These studies are relevant when determining erosion yield using the recession depth technique. Also characterized was the hygroscopic nature (or moisture absorption) of each polymer, which is relevant to determining erosion yield using the mass loss technique. Uncertainties of the erosion yield as determined by both the mass loss and recession depth techniques were reviewed and compared. Studies and experimental development continues on the PEACE experiment. It will fly for approximately two weeks on board a space shuttle orbiter sometime in the future.

VI. REFERENCES

1. J. A. Dever, "Low Earth Orbital Atomic Oxygen and Ultraviolet Radiation Effects on Polymers," NASA TM 103711, February 1991.
2. K. K. de Groh, B. A. Banks, G. W. Clark, A. M. Hammerstrom, E. E. Youngstrom, C. Kaminski, E. S. Fine & L. M. Marx, "A Sensitive Technique Using Atomic Force Microscopy to Measure the Low Earth Orbit Atomic Oxygen Erosion of Polymers," NASA TM-2001-211346, 2001.
3. B. Banks, S. Rutledge, E. Sechkar, T. Stueber, A. Snyder, Kim de Groh, C. Haytas & D. Brinker, "Issues and Effects of Atomic Oxygen Interactions with Silicone Contamination on Spacecraft in Low Earth Orbit," NASA TM-2000-210056, 2000.
4. J. Visentine, W. Kinard, R. Pinkerton, D. Brinker, D. Sheiman, B. Banks, J. Zwiener, K. Albyn, T. Farrell, S. Hornung, T. See, "MIR Solar Array Return Experiment," Presented at the 37th AIAA Aerospace Sciences Meeting, January 11-14 1999, Reno, NV, AIAA 99-0100, 1999.
5. J. Kleiman, Z. Iskanderova, B. A. Banks, K. K. de Groh, E. A. Sechkar, "Prediction and Measurement of the Atomic Oxygen Erosion Yield of Polymers in Low Earth Orbital Flight," Proceedings of the 8th International Symposium on Materials in a Space Environment, June 5-9, 2000, Arachon, France.
6. K. K. de Groh, B. A. Banks, A. Hammerstrom, E. Youngstrom, C. Kaminski, L. Marx, E. Fine, J. D. Gummow, and D. Wright "MISSE PEACE Polymers: An International Space Station Environmental Exposure Experiment," NASA TM-2001-211311 and AIAA-2001-4923, 2001.

7. ASTM E 595-84 Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment, *Annual Book of ASTM Standards*, 1984.
8. MISSE home page: <http://misse1.larc.nasa.gov/index.html>, 2001.
9. B. A. Banks, K. K. de Groh, E. Baney-Barton, E. A. Sechkar, P. K. Hunt, A. Willoughby, M. Bemer, S. Hope, J. Koo, C. Kaminski & E. Youngstrom, "A Space Experiment to Measure the Atomic Oxygen Erosion of Polymers and Demonstrate a Technique to Identify Sources of Silicone Contamination," NASA TM-1999-209180, 1999.

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